Experimental report

03/09/2021

Proposal: **EASY-714** **Council:** 4/2020

Title: COMPONENT DYNAMICS IN POLYMERIC MIXTURES OF INDUSTRIAL INTEREST

Research area: Soft condensed matter

This proposal is a new proposal

Main proposer: **Numera SHAFQAT**

Experimental team:

Local contacts: Lucile MANGIN-THRO

Samples: SBR/PS

Instrument	Requested days	Allocated days	From	To
D7	24	24	27/09/2020	28/09/2020

Abstract:

Blends of SBR and PS are of interest for the tire industry. Here we want to use them also as model systems to investigate basic properties of dynamically asymmetric mixtures.
With these experiments we want to selectively investigate the component dynamics in blends where PS component will be deuterated and the SBR protonated to follow SBR and the inverse labeling to follow PS with one day beamtime. Different concentration (ɸSBR=50 and 80) shall be explored in the range between 200 and 300K.

COMPONENT DYNAMICS IN POLYMERIC MIXTURES OF INDUSTRIAL INTEREST

Easy proposal EASY-714. Experimental report

D7 - 27/09/2020 to 28/09/2020 - Local contact: Mangin-Thro Lucile.

Blends of SBR (styrene-butadiene rubber) and PS (polystyrene) are of interest for the tire industry. Here we want to use them also as model to investigate basic properties of dynamically asymmetric mixtures. With these experiments we want to selectively investigate the component dynamics in blends where PS component will be deuterated and the SBR protonated to follow SBR hydrogen self-motions in the blend and the inverse labeling to follow the PS component.

Samples where the SBR is deuterated and the PS is protonated and samples where the SBR is protonated and PS is deuterated were prepared. Protonated and deuterated styrene-butadiene rubber (hSBR and dSBR) were synthesized by anionic polymerization by the Michelin Company. The protonated and deuterated polystyrene (hPS and dPS) were purchased from Polymer Source. Since the blend is immiscible for high-M_w PS, we have considered oligomers of M_n= 500 g/mol. Table 1 shows the molecular weights, glass transition temperatures T_gs as determined by differential scanning calorimetry (DSC) and densities of the pure components. Two different compositions (ϕ_{SBR}=50, 80%) have been explored: 80dSBR/20hPS, 80hSBR/20dPS, 50dSBR/50hPS and 50hSBR/50dPS.

sample	M_n (g/mol)	PDI	T_g (K)	d (g/cm^3)
hSBR	69900	1.09	208	0.94
dPS	500	1.12	260	1.07
dSBR	38100	1.13	210	1.06
hPS	500	1.2	273	0.999

Table 1. Molecular weights, Polydispersities, Glass-Transition Temperatures and Densities of the Homopolymers.

Samples were filling flat aluminium sampleholders, and the thicknesses were calculated to yield a transmission of 90%. The samples were investigated by IN13. As complementary experiment, by means of D7 we measured the ratio between coherent and incoherent contributions to the differential scattering cross-section to determine the range of Q where we could safely consider that the IN13 results mainly reflected incoherent dynamics of the hydrogenated blend component.

D7 experiments were carried out where the incident neutron wavelength (λ) was set to 4.89 Å to cover a Q-range from 0.15 to 2.5 $Å^{-1}$. A vanadium sheet was used to calibrate the detector efficiency. For comparison reasons, the same experiment has been done in the homopolymer dPS as reference sample.

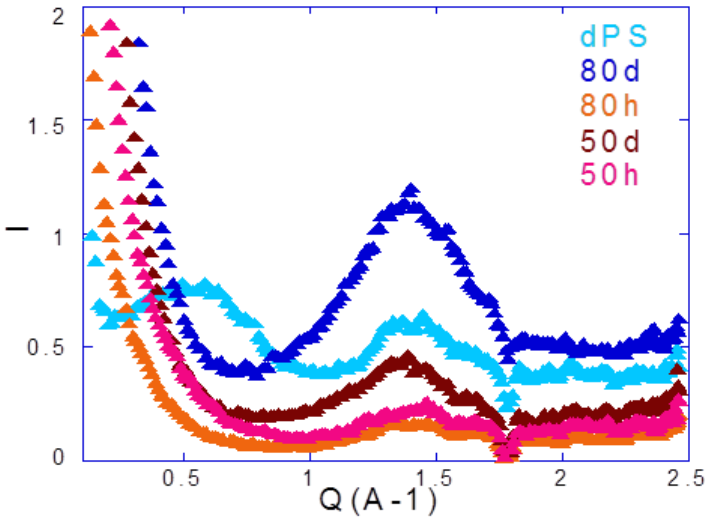

Figure 1. Ratio between the coherent and incoherent differential scattering cross section obtained from D7 experiments of diffraction with polarization analysis on the pure homopolymer dPS and the blends with ϕ_{SBR}= 0.8 and 0.5.

The D7 results shown in Fig. 1 reveal that below Q ≈ 0.3$Å^{-1}$ the scattered intensity is dominated by concentration fluctuations. At higher Q-values, incoherent scattering is much higher than coherent scattering, with exception of the sample rich in dSBR (80d) in the neighbourhood of the main structure factor peak (at about 1.3$Å^{-1}$). This information is of importance when evaluating and interpreting IN13 results on these samples.